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STABLE AQUEOUS DISPERSIONS OF QUATERNARY AMMONIUM COMPOUNDS AND IMIDAZOLINE DERIVATIVES

[Stabile waesserige Dispersionen von quaternaeren Ammoniumverbindungen und imidazolin-Derivaten]

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Stable Aqueous Dispersions of Quaternary Ammonium Compounds and Imidazoline Derivatives

The invention concerns aqueous dispersions of quaternary ammonium compounds and/or imidazoline derivatives having limited water solubility, which are stabilized with regard to their dispersion state by adding water-in-oil dispersions of water-swellable, cationically active, crosslinked polymers. The stabilizing acting water-in-oil dispersions are comprised by 30 to 70 percent by weight of a cationically active homopolymers or copolymer, 20 to 50 percent by weight of a hydrophobic organic fluid, 0.6 to 10 percent by weight of a water-in-oil emulsifier, 0 to 10 percent by weight of a wetting agent, and residual water to complete 100%. The stabilizing-acting cationic crosslinked polymers are preferably obtained by means of emulsion polymerization. The invention concerns also the use of these stabilizing aqueous dispersions as fabric and clothing softeners.

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Specification

[1] Numbers in the margin indicate pagination in the foreign text.

The invention concerns aqueous dispersions of quaternary ammonium compounds and imidazoline derivatives, which are stabilized by means of water-in-oil dispersions of cationic, crosslinked, water-swellable polymerizates.

Quaternary ammonium or cyclic amidine compounds of fatty chemical raw materials, which are used, for example, as active components in fabric and clothing softener formulations, have frequently only a limited water solubility and tend therefore to separate when they are introduced into the water and their solubility limit is exceeded. This leads not only to difficulties during storage, but also to more inaccurate or defective dosages when they are used. There have therefore been multiple attempts at finding out with which agents can be obtained castable or pasty dispersions that remain stable during storage. WO 91/12364, for example, describes aqueous preparations, which store well and contain an active ingredient combination based on condensation products of carbonic acids or carbonic acid derivatives with hydroxy alkyl polyamines and quaternary ammonium compounds. Also part of the state of the art is the addition at ambient temperature of solid cationic ammonium or cyclic amidine compounds of low viscous, water-soluble alcohols, such as methanol, ethanol or isopropanol. Also ethylene glycol, propandiol-1,2 and the homologous high

molecular polyalkylene glycol ethers found use, because to their high flammability point. It can be evaluated as disadvantageous that these solvents are not only combustible to a great extent and may migrate within plastic packaging materials, but also that they have a destabilizing effect on the dispersion if they are further diluted. A stabilizer for aqueous cationic dispersions, whose flammability point is over 100°C and whose properties allows any kind of plastic packaging is therefore desirable.

It is an object of the invention, therefore, to further improve the effect of the known stabilizing additives.

This object is attained in accordance with the invention with weakly crosslinked water-swellable copolymerizates based on cationic monoethylenic unsaturated carbonic acid derivatives.

Polymers that cause an increase in viscosity due to the presence of water-swellable particles in water or aqueous solutions are used in many fields of technology, cosmetics and pharmacy. Naturally occurring substances, such as alginates, guar flour and starch of modified or chemically modified type are being increasingly replaced by synthetic polymers, wherein polymers of acrylic compounds, among others, are used.

Three-dimensional networks can be created by using multifunctional compounds to a lesser extent, so that the

products can absorb greater amounts of water, without being transferred completely into the dissolved state. They remain only still water-swellable, even though they contain in general only water-soluble components.

In particular water-in-oil emulsion polymerizates of these acrylic compounds produced by inverse emulsion polymerization offer advantages due to the easy way in which they are introduced into water or aqueous media. The production of water-soluble polymers of this substance class by means of emulsion polymerization is described in United States patent 3,284,393 and in DE-OS 22 26 143. Since these products represent water-in-oil dispersions, which contain the polymer in emulsified "water droplets," a reverse emulsifier for dispersing the external oily phase pursuant to DE-OS 22 26 143 must be used in order to achieve the solution in water. The emulsion type reverses afterward to oil-in-water. According to DE-OS 24 31 794, the wetting agent can already be contained in the dispersion, whereby it becomes self-inverting. Crosslinked, water-insoluble or water-swellable polymers of non-ionogenic or anionic monomers from the acryl series are described, for example, in the patent publication GB 20 07 238 and the United States patent 4,554,018.

Water-soluble, non-crosslinked, inverse emulsion polymerizates from the acryl series with cationic monomers are described in DE-OS 27 56 697, whose production and mode of application is adjusted, however, to their special application purpose as flocculants. As thickeners for non-aqueous solutions are mentioned in the European application weakly crosslinked, inverse emulsion polymerizates with cationic (meth)acrylic acid esters. Corresponding water-in-oil dispersions of water-swellable, weakly crosslinked, cationic polymers are proposed in DE 37 30 781 C2 for the production of printing pastes for textile printing. N-substituted (meth)acrylic acid [illegible] due to the stronger hydrophilia of the amide groups in contrast with the ester function and [illegible] are thus considered particularly suitable due to their water swellability. At the same time, these amides have a better hydrolysis stability, which is important for long-term applications in aqueous solution.

Since the viscosity of ionic polymerizates decreases considerably due to electrolyte addition, it is reported in EP 0 186 361, that the thickening effect of partially ionic, preferably anionic, weakly crosslinked polymers in electrolyte-containing media can be improved by adding ionic tensides, if these are soluble to a greater extent in the oily phase of the

water-in-oil dispersions than in the aqueous phase. While the anionic polymers and anionic tensides are described extensively and in detail in this publication, the cationic products are taken into consideration by merely naming their application possibilities, without mention of experimental findings.

It is an object of the invention, therefore, to use water-in-oil dispersions of water-swellable, predominantly cationic, crosslinked polymers for stabilizing aqueous dispersions of quaternary ammonium compounds and imidazoline derivatives, as used, for example, in so-called fabric and clothing softeners. These aqueous dispersions contain as customary active ingredients, for example, N,N-distearyl-N,N-dimethyl ammonium chloride (DSDMAC), imidazoline derivatives (see among others United States patent 4,762,685, EP-A 0 199 383), quaternary fatty acid esters of triethanol amine (so-called esterquats, see among others United States patent 4,830,771) or

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2,3-dihydroxy propyl-1-trimethyl ammonium salt derivatives (DE-OS 27 28 841).

Ammonium compounds having the formula $[R_1R_2R_3R_4N]^+X^-$ are used in general, in which:

R_1 = an alkyl group with 1 to 4 carbon atoms, in particularly methyl,

R_2 = an alkyl or alkylene group with 1 to 22 carbon atoms, or a hydroxyalkyl group with 2 to 4 carbon atoms,

R_3 = an alkyl or alkylene group with 1 to 22 carbon atoms, an acyloxy alkyl group, whose acyl radical is derived from saturated or unsaturated, linear or branched fatty acids with 10 to 22 carbon atoms, and whose oxyalkyl group is the oxyethyl or oxypropyl or oxyisopropyl group,

R_4 = R_3 and R_2, and

X^- = an anion.

The surface floating and/or bottom sediment formations that occur generally in unstable suspensions during storage can be reduced by means of viscosity increases, but cannot be eliminated. If the stabilizers according to the invention are used, the dispersion state is improved to such an extent that the homogeneity during storage and handling can be ensured.

The water-in-oil dispersions used for the stabilization consist of:

A) 30 to 70 percent by weight of cationically active homopolymers or copolymer,

B) 20 to 50 percent by weight of hydrophobic, organic fluid,

C) 0 to 10 percent by weight of oil-in-water emulsifier,

D) if required 0.5 to 10 percent by weight of a wetting agent, and

E) a residual amount of water.

The sum of the components (A) through (E) produces 100 percent by weight.

The production of the homopolymers or copolymers occurs by polymerization of a monomer mixture of

a) at least 50 percent by weight of one or several cationic monomers having the general formula:

$[CH_2=C(R_1)-CO-X-(CH_2)_n-C(R_2R_3)-CH_2N(R_4R_5R_6)]^+A^-$,

in which

R_1 = hydrogen or methyl,

R_2, R_3 = hydrogen or identical or different alkyl radicals with 1 to 4 C atoms,

R_4, R_5, R_6 = identical or different alkyl radicals with 1 to 4 C atoms,

X = -O- or -NH-, and

A = anion of an organic or inorganic acid, preferably hydrochloric acid or methyl sulfuric acid,

b) 0 to 40 percent by weight of one or several non-ionic, water-soluble monomers, such as acrylamide and methacryl amide, allyl alcohol, allyl polyethylene glycol ether, and hydroxy ethyl methacrylate,

c) 0 to 40 percent by weight of one or several anionic, water-soluble monomers, such as acrylic acid, methacrylic acid, (meth)acrylamidomethyl butanic acid, and/or acrylamido-2-methyl propanosulfonic acid, and/or their salts,

d) 0.001-5.0 percent by weight of a polyfunctional monomer.

As cationic monomers are used quaternized dimethyl aminoethyl (meth)acrylate, quaternized dimethyl aminopropyl (meth)acrylamide, quaternized dimethyl amino-2,2-dimethyl propyl (meth)acrylamide, diallyl dimethyl ammonium chloride, diallyl ethyl ammonium chloride, and their mixtures among each other. The quaternization is preferably carried out with methyl chloride, sodium chloroacetate, dimethyl sulfate, and dimethyl carbonate.

As hydrophobic organic fluid can be used: aromatic and aliphatic linear, branched, and cyclic hydrocarbons, whose boiling points at a pressure of 1013 hPa are within the range of 110 to 380°C. To these belong n-paraffins and isoparaffins, distillates of paraffinic, napthene-based, and aromatic petroleum, as well as toluol, xylol and mesitylene. Linear and branched fluid esters of natural or synthetic origin can also be used as oily phase.

In order to prepare the polymerization and production of water-in-oil emulsions containing monomers are utilized known oil-soluble emulsifiers with low HLB value, such as partial esters and glycerin, diglycerin and polyglycerin, sorbitol, sorbitan, and additives of alkylene oxides, such as ethylene oxide and propylene oxide, on higher linear and branched alcohols, fatty acids or nonyl phenol. Also so-called polymeric emulsifiers can be used alone or in mixture, as is described, inter alia, in United States patent 4,786,681.

As crosslinking monomers are utilized multiply unsaturated vinyl, acryl, or allyl compounds, as well as compounds with several epoxy groups. Suitable compounds are, for example, methylene-bis-acryl amide as well as quaternized triallyl amine, in particular tetraallyl or triallyl methyl ammonium chloride. The crosslinking can take place during the polymerization or also thereafter. For a post-crosslinking are particularly suitable water-soluble epoxy compounds derived from polyvalent alcohols.

The technique of polymerization with emulsion formation, homogenization of the mixture, and initiation of the polymerization is known. It takes place preferably adiabatically, but can also occur at a preselected temperature. After the polymerization has ended, the water-in-oil dispersion

can be directly introduced into the aqueous, cationic dispersion of quaternary ammonium or cyclic amidine compounds.

The water-in-oil dispersions can be utilized for stabilizing aqueous dispersions with limited water-soluble, quaternary ammonium salts, such as dimethyl distearyl ammonium chloride, dimethyl dilauryl ammonium chloride, and quaternary esters, for example, N-methyl-N,N-bis[2-(acyloxy)ethyl]-N-2-hydroxy ethyl ammonium methosulfate or N,N,N-trimethyl-N-[1,2-di(acyloxy)propyl]-ammonium chloride, or imidazoline derivatives based on fatty acids, for example, 1-[(fatty acyloxy)ethyl]-2-alkyl imidazoline.

The quaternary ammonium salts or imidazoline derivatives having a limited solubility in water are usually available as aqueous suspensions/dispersions, for example, with solids contents of 5 percent by weight. The solids content is otherwise not critical.

The application concentrations fluctuate between 0.01 and 10 percent by weight, preferably between 0.1 and 5 percent by weight, especially between 0.2 and 2 percent by weight, depending on the desired stabilization of the dispersion. In addition can also be utilized viscosity-increasing non-ionic or cationic substances.

It was surprisingly discovered that the stability of aqueous cationic dispersions, in particular of limited water-soluble quaternary ammonium salts, is clearly increased with the aid of water-in-oil dispersions of cationically crosslinked water-swellable polymers.

The invention is explained in more detail with reference to the following examples:

In the description of the following examples are utilized abbreviations having the following meanings:

DMAEA = dimethyl aminoethyl acrylate

DMAEMA = dimethyl aminoethyl methacrylate

DIMAPA = dimethyl aminopropyl acrylamide

TEMAPA = N,N,N,2,2-tetramethyl aminopropyl acrylamide

AMPS = 2-acrylamido-2-methyl-propane sulfonic acid

TAMAC = triallyl methyl ammonium chloride

ABAH = azo-bis-(2-amidinopropane hydrochloride)

AIBN = azo-isobutyric acid dinitrile

EDTA = ethylene diamine tetraacetic acid

TBHP = tert. butyl hydroperoxide

EO = ethylene oxide

KWST = isoparaffinic hydrocarbon having a boiling range of 210-250°C and a density of 0.79 g/cm^3.

All data in percents mean percent by weight.

Example 1

a) Production of the dispersion of a polymer of 80% of DMAEA-CH_3Cl and 20% acrylamide with 40% active substance (wS).

160 g of DMAEA-CH_3Cl and 40 g of acrylamide are dissolved in 165 g of water. Thereafter, the pH value is set to 4.5 with phosphoric acid and 0.4 g of EDTA are added. After the solution of the complex builders has ended, the solution is poured while stirring into a mixture of 120 g of KWST, 10 g of sorbitan monoisostearate, and 0.62 g of TAMAC and homogenized with a usual household stirring stick (viscosity of the emulsion 4200 mPa·s, Brookfield RVT, MK II/20 at 20°C). The dissolved oxygen is eliminated by blowing in nitrogen with a simultaneous heating of the emulsion to 60°C. Nitrogen is reintroduced then for approx. 30 minutes and the polymerization is then started by adding 0.11 g of AIBN dissolved in 4 g of KWST. The temperature increases approximately to 102°C by means of the adiabatically controlled exothermal reaction. It is stirred for approximately 1 hour more and then mixed with 10 g of EO nonyl phenol.

b) With the aid of a fast stirring are introduced 5 g of this dispersion into 1 l of 5% solution of methyl quaternized digital fatty acid triethanol amine esters, whereupon a dispersion that is stable during storage and does not show any changes over a time period of 6 weeks is produced.

Example 2

a) Production of a dispersion of a polymer of 80% of DIMAPA-CH_3Cl and 20% acrylamide with 40% active substance (wS).

160 g of DIMAPA-CH_3Cl and 40 g of EDTA are dissolved in 170 g of water. Thereafter, the pH value is set to 4.5 with hydrochloric acid and 0.4 g of EDTA are added. After the complex builders have completely dissolved, the solution is poured while stirring into a mixture of 115 g of KWST, 10 g of glycerin monooleate, and 0.62 g of TAMAC and homogenized with a usual household stirring stick (viscosity of the emulsion 4500 mPa·s, Brookfield RVT, MK II/20 at 20°C). After the dissolved oxygen has been eliminated with nitrogen, the polymerization is started at 60°C by adding 0.11 g of AIBN dissolved in 4 g of KWST. A temperature increase to approximately 102°C follows. It is stirred for approximately 1 hour more and then mixed with 10 g of EO nonyl phenol.

b) By stirring 5 g of this dispersion into 1 l of 5% solution of distearyl dimethyl ammonium chloride in water is produced a dispersion that is stable during storage and whose condition does not change over a time period of 6 weeks.

Example 3

a) Production of a dispersion of a polymer of 75% of DIMAPA-CH_3Cl and 12.5% acrylamide and 12.5% acrylic acid with 40% active substance (wS).

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150 g of DIMAPA-CH_3Cl, 25 g of acrylamide, and 40 g of EDTA are dissolved in 170 g of water, and 25 g of acrylic acid are added. Thereafter, the pH value is set to 4.5 with sodium hydroxide and the solution is poured while stirring into a mixture of 115 g of KWST, 10 g of sorbitan monoisostearate, and 0.62 g of TAMAC. It is homogenized with a usual household mixing stick, flushed with nitrogen, and then started with 0.0275 g of AIBN dissolved in 4 g of KWST.

b) After the polymerization has concluded, 5 g of this dispersion are added into 1 l of 5% solution of methyl quaternized ditallow fatty acid triethanol amine ester, whereupon a dispersion that is stable during storage and whose condition does not change over a time period of 6 weeks is produced.

Example 4

a) Production of a dispersion of 92% of DMAEMA-CH_3Cl and 8% acrylamide with 50% active substance (wS).

230 g of DMAEMA-CH_3Cl and 20 g of acrylamide are dissolved in 110 g of water and 0.5 g of EDTA, and 0.6 g of ABAH are

added. The pH value is set to 0.6 with concentrated hydrochloric acid. The formation of the emulsion occurs as in Example 1 with 120 g of KWST and 10 g of sorbitan monoisostearate (viscosity: 1400 mPa·s at 20°C). It is catalyzed with 6 mg of TBHP and 12 mg of sulfur dioxide at 20°C, whereupon a peak temperature of 72°C is reached within 150 minutes. It is stirred for approximately 1 hour more and then mixed with 10 g of EO nonyl phenol.

b) 5 g of the dispersion in 1 l of 5% solution of distearyl dimethyl ammonium chloride at 20°C produce a suspension that is stable during storage and does not have a tendency to settle on the bottom or to float on the surface.

Example 5

a) Production of a dispersion of 60% of DMAEA-CH_3Cl, 37% of acrylamide, and 3% of AMPS with 46% active substance (wS).

160 g of DMAEA-CH_3Cl, 36 g of acrylamide, and 19 g of AMPS are dissolved in 114 g of water, and after adjusting the pH value to 4.5 with hydrochloric acid, it is poured while stirring 100 g of KWST, 10 g of diglycerin sesquioleate, 0.8 g of TAMAC, and 0.2 g of EDTA. It is homogenized, flushed with nitrogen, and the polymerization is started with 1 g of AIBN.

b) 5 g of the dispersion produce 1 l of a 5% suspension of distearyl dimethyl ammonium chloride that is stable during storage and does not have a tendency to inhomogeneity.

Further Examples with Different Compositions of the Polymer Products

a) The production of the dispersions corresponds to the mode of procedure of the previous descriptions. The polmerizations were started at ambient temperature.

Quantity Ratios of Used Raw Materials (g)

Example	6	7	8	9	10
Water	139	139	164	281	164
Acrylic acid		15	40	80	16
Acrylamide	40	25			25
DIMAPA quat.			175	120	175
TEMAPA quat.	200	200			
EDTA	0.2	0.2	0.2	0.2	0.2
TAMAC	016	0.6	0.6	0.6	0.6
ABAH	0.3	0.3	0.3	0.3	0.3
pH value	4.5	4.5	4.5	4.5	4.5
KWST	110	110	110	110	110
Sorbitan monoisostearate	10	10	10	10	10
TBHP (0.2%)	1	1	1	1	1

6-EO-nonyl phenol	10	10	10	10	10

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Percentage of Polymers in Composition (Percent by Weight)

Example	6	7	8	9	10
Acrylic acid		6	19	40	10
Acrylamide	17	10			
DIMAPA quat.			81	60	81
TEMAPA quat.	83	84			

All of the dispersions listed in the table produced suspensions that are stable during storage and do not have a tendency to settling on the bottom or floating on the surface with an addition of 5 g of 5% suspensions of N-methyl-N,N-bis[2-C16/18-acryloxy)-ethyl]-N-(2-hydroxy ethyl)-ammonium methosulfate.

Patent Claims

1. Aqueous dispersions of quaternary ammonium compounds and/or imidazoline derivatives having limited water solubility, which are stabilized with regard to their dispersion state by adding water-in-oil dispersions of water-swellable, cationically active, crosslinked polymers.

2. The aqueous dispersions of quaternary ammonium compounds and/or imidazoline derivatives having limited water solubility of claim 1, wherein the water-in-oil dispersions of water-swellable, cationically active, crosslinked polymers are obtained from the components

   A) 30 to 70 percent by weight of a cationically active homopolymers or copolymer,

   B) 20 to 50 percent by weight of hydrophobic, organic fluid,

   C) 0 to 10 percent by weight of oil-in-water emulsifier,

   D) if required 0.5 to 10 percent by weight of a wetting agent, and

   E) a residual amount of water,

   in which the sum of the components (A) through (E) produces 100 percent by weight.

3. The aqueous dispersions of quaternary ammonium compounds and/or imidazoline derivatives having limited water solubility of claims 1 and 2, wherein the water-swellable, cationically active, crosslinked polymers are obtained as homopolymers or copolymers by inverse emulsion polymerization of

a) 50 to 100 percent by weight of a monomer having the general formula

$[CH_2=C(R_1)-CO-X-(CH_2)_n-C(R_2R_3)-CH_2-N(R_4R_5R_6)]^+A^-$,

in which

R_1 = hydrogen or methyl,

R_2, R_3 = hydrogen or identical or different alkyl radicals with 1 to 4 C atoms,

R_4, R_5, R_6 = identical or different alkyl radicals with 1 to 4 C atoms,

X = -O- or -NH-, and

A = anion of an organic or inorganic acid, preferably hydrochloric acid or methyl sulfuric acid,

b) 0 to 40 percent by weight of one or several non-ionic, water-soluble monomers, such as acrylamide and methacryl amide, allyl alcohol, allyl polyethylene glycol ether, and hydroxy ethyl methacrylate,

c) 0 to 40 percent by weight of one or several anionic, water-soluble monomers, such as acrylic acid, Methacrylic acid, (meth)acrylamidomethyl butanic acid, and/or acrylamido-2-methyl propanosulfonic acid, and/or their salts,

d) 0.001-5.0 percent by weight of a polyfunctional crosslinking monomer,

in which the sum of the components (a) through (d) produces 100 percent by weight.

4. The aqueous dispersions of one of the claims 1-3, wherein 0.01-10 percent by weight of a water-in-oil dispersion of water-swellable, cationically active, crosslinked polymers with reference to the dispersion to be stabilized are added.

5. A use of water-in-oil dispersions, whose aqueous phase contains water-swellable, cationic, crosslinked polymers for the stabilization of aqueous dispersions of quaternary ammonium or cyclic amidine compounds having limited water solubility.

6. The use of water-in-oil dispersions of claim 4, wherein said dispersions are obtained from the components

   A) 30 to 70 percent by weight of a cationically active homopolymers or copolymer,

   B) 20 to 50 percent by weight of hydrophobic, organic fluid,

   C) 0.5 to 10 percent by weight of oil-in-water emulsifier,

   D) 0 to 10 percent by weight of a wetting agent, and

   E) a residual amount of water,

in which the sum of the components (A) through (E) produces 100 percent by weight.

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7. The use of water-in-oil dispersions of claims 4 and 5, wherein the cationically active homopolymers or copolymer is obtained by inverse emulsion polymerization of

a) 50 to 100 percent by weight of a monomer having the general formula

$[CH_2=C(R_1)-CO-X-(CH_2)_n-C(R_2R_3)-CH_2-N(R_4R_5R_6)]^+A^-$,

in which

R_1 = hydrogen or methyl,

R_2, R_3 = hydrogen or identical or different alkyl radicals with 1 to 4 C atoms,

R_4, R_5, R_6 = identical or different alkyl radicals with 1 to 4 C atoms,

X = -O- or -NH-, and

A = anion of an organic or inorganic acid, preferably hydrochloric acid or methyl sulfuric acid,

b) 0 to 40 percent by weight of one or several non-ionic, water-soluble monomers, such as acrylamide and methacryl amide, allyl alcohol, allyl polyethylene glycol ether, and hydroxy ethyl methacrylate,

c) 0 to 40 percent by weight of one or several anionic, water-soluble monomers, such as acrylic acid, Methacrylic acid, (meth)acrylamidomethyl butanic acid, and/or acrylamido-2-methyl propanosulfonic acid, and/or their salts,

d) 0.001-5.0 percent by weight of a polyfunctional crosslinking monomer,

in which the sum of the components (a) through (d) produces 100 percent by weight.

8. A use of aqueous dispersions of quaternary ammonium compounds and/or imidazoline derivatives having limited water solubility, stabilized with water-in-oil dispersions of the claims 1 to 3 as fabric or clothing softeners.

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